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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
C09K 3/10, B29C 59/02, C08J 3/075, H01B 7/28, H02G 15/08

A1

(11) International Publication Number: **WO 97/19143**

(43) International Publication Date: 29 May 1997 (29.05.97)

(21) International Application Number: PCT/GB96/02871

(22) International Filing Date: 21 November 1996 (21.11.96)

(30) Priority Data:

9523960.4	23 November 1995 (23.11.95)	GB
9610000.3	14 May 1996 (14.05.96)	GB

(81) Designated States: JP, KR, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

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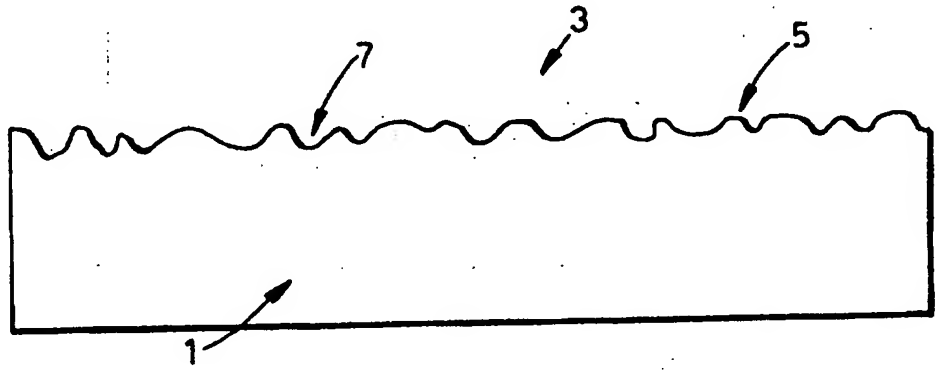
(54) Title: GEL SEALANTS AND ARTICLES



(57) Abstract

Gel sealants and articles having deliberately roughened exterior surface to facilitate handling (reduces tack) and/or enhance separability and appearance of gel surfaces. The roughening may be achieved by deliberately induced surface melt fracture during melt processing (extruding) of thermoplastic gels, or by moulding or casting the gels on rough surfaces such as abrasive sheets to produce a pitted surface. Preferred gel materials are highly-oil-extended styrene-hydrogenated alkylene-styrene triblock copolymer gels.

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GEL SEALANTS AND ARTICLES

The present invention relates to gel sealant, e.g. for sealing containers, especially containers for containing one or more electrical connections, electrical or electronic devices, optical fibres, or optical devices; and also relates to articles comprising gel, e.g. thermoplastic gel tapes, sheets and profiles, or devices incorporating such gel sealants or articles.

Various gel sealant materials are known, for example thermoplastic gels described in International Patent Applications WO-A-88/00603 (RK308), WO-A-90/05166 (RK403), WO-A-93/05113 (RK451), WO-A-93/23472 (RK469), and WO-A-94/18273 (RK472), the entire disclosures of each of which are incorporated herein by reference. Articles comprising gel are useful for many sealing purposes, especially the aforementioned gel tapes, sheets, and profiles, which are described for example in our International Patent Applications WO-A-9609483 (RK508), WO-A-9618836 (B288), PCT/GB96/02170 (RK532), and PCT/GB96/02000 (RK533).

The present invention ingeniously controls the surface tack and separability of such gel sealants and articles to facilitate handling and/or enhance appearance, without significantly detracting from the eventual end use properties of the gel.

The invention accordingly provides an article or object comprising a body of gel material having a deliberately roughened exterior surface. It will be understood that the roughened exterior surface of the gel may carry a regular or random pattern of roughening. Regular patterns might include those obtained by casting the gel on a mesh or grid. Random patterns may be preferred, for example those produced by deliberate surface melt fracture or by moulding the gel on a sheet abrasive material. The roughening will extend, preferably substantailly uniformly, over a useful area of the article or object, frequently over a majority, or substantially the whole, of the gel surface which is exposed to contact in subsequent storage or use.

One aspect of the invention provides a melt-extruded thermoplastic gel article having a tack-reducing degree of surface melt fracture.

Melt fracture is usually regarded as a defect, and extruder operators normally take considerable trouble to prevent surface melt fracture in the extruded article. It is contrary to normal practice to make the present advantageous use of the melt fracture "defect" to control tack while avoiding the known disadvantages of talc dusting or other tack-reducing additives.

It will be understood that references herein to a tack-reducing degree of surface melt fracture mean that the tackiness of the gel is reduced in comparison with the same article when produced substantially free of surface melt fracture.

The desired degree of melt fracture may be induced by running the extruder with the exit tip region containing the extrusion die at a lower-than-normal temperature, possibly involving cooling of the die head. The exact temperature will vary depending on the gel material being extruded, but will readily be determined by simple trial variations until the desired surface finish is produced. Preferably, the degree of surface melt fracture is at least sufficient to provide the article with a visually-observable matt surface finish, more preferably a "sharkskin" melt-fractured surface finish, which is well recognised (as a defect) in the field of plastics extrusion.

The invention also provides a method of making a melt-extruded thermoplastic gel article, wherein, during the melt extrusion process, surface melt fracture of the gel is induced to a degree which reduces the tack of the finished article, preferably to provide the aforementioned visually-observable surface finish.

The degree of melt-fracture tends to be self-limiting in that it may be increased until the extruded gel surface becomes unacceptably rough, or, in extreme cases, the extruded material fails to cohere and/or major cracks or voids appear in it.

The gel article and/or the gel material itself and methods for characterising the gels may be as generally described in any of the aforementioned International applications, the disclosures of all of which are incorporated herein by reference. Preferred articles according to this aspect of the present invention are those wherein the surface-melt-fractured gel comprises a [hard block-elastomeric block-hard block] triblock copolymer whose elastomeric mid-block is extended with at least 200 parts by weight of extender fluid per 100 parts by weight of the triblock copolymer. Especially preferred are those wherein the triblock copolymer is a [styrene-hydrogenated alkylene-styrene] triblock copolymer, preferably having a hydrogenated alkylene mid-block comprising ethylene/propylene and/or ethylene/butylene units, examples of which gels using triblock copolymers available under the trade marks "Kraton" and "Septon" are known from the references listed above.

A specific embodiment of this aspect of the present invention will now be described by way of example. A gel material was formed by melt blending 24% by weight of Septon 2006 (Trade Mark) styrene-ethylene/propylene-styrene triblock copolymer and 2% by weight of known antioxidant with 74% by weight of Fina A360B extender oil and extruding the gel as a ribbon or tape from a Francis Shaw 63.5mm (2.5 inch) extruder using a 8mm x 5mm rectangular die hole and a polyethylene-type screw having a 25:1 length:diameter ratio. The extruder barrel temperatures, from the feed hopper to the die heaters, were 165, 195, 210, 230, and 230°C. The die heater zones were set at 230, 230, and 265°C (closest to the die face), which induced on the extrudate, at the line speed and output delivered by the chosen screw, a melt-fractured surface within the preferred range intermediate between the minimum visually-observable matt finish and coarse "sharkskin" melt fracture.

For comparison, the die lip temperature (the last surface touched by the gel leaving the die) may be increased and/or the extrusion speed decreased to a level at which a full gloss surface is produced on the extruded gel tape. The resulting full-gloss tapes are discernably tackier to the touch than the deliberately melt-fractured surface of the product described above.

In this aspect of the invention, it may be adviseable to limit the degree of surface melt fracture to avoid any unacceptable tendency towards crack propagation, especially if the gel is to be placed under tension in its end use.

In a second aspect of the present invention, it has been discovered that, by providing gel sealant objects with a surface texture as defined below, a number of advantages may be obtained.

According to this second aspect, the present invention provides an object comprising gel sealant, a gel surface of which has a pitted texture which is visible to the naked (i.e. unassisted) eye.

Gel sealant materials are normally put under compression in use, e.g. in order to enhance the tightness of the seal produced by the gel sealant. One advantage of gel sealant objects according to the invention is that when the gel sealant is put under compression, e.g. by means of another object in contact with the pitted texture gel surface, the gel surface normally has a reduced tendency to be damaged (e.g. torn, cut or chopped) in comparison to a similar gel sealant object in which the contacted gel surface does not have such a pitted texture.

This second aspect of the invention is not limited by any theory seeking to explain its advantages, but it is believed that this reduced tendency to become damaged by compressive contact may be due, at least in part, to the increased surface area provided by the pitted texture of the surface of the gel. It is thought that the textured surface, having an increased surface area compared to a non-textured surface, is required to stretch less (compared to a corresponding non-textured surface) when placed under compressive contact. Therefore the gel sealant at the textured surface (and preferably also near the surface) is normally further from its elastic limit, and thus retains a greater degree of resilience, when placed under compression, with which to withstand the stresses produced by the compressive contact. In some situations, gel sealant may be placed under localized extreme stresses, for example where the gel is required to seal around electrical contacts (e.g. in a telecommunications terminal block). By providing

the gel with a pitted surface texture according to the invention, such localized stresses may be reduced, thus reducing the likelihood of damage to the gel.

Another advantage of the pitted surface texture of gel sealant objects according to this second aspect of the invention is that it may reduce the tackiness of the gel surface compared to a non-textured surface. Thus the release of the gel sealant from an article, or indeed from other gel sealant, may be made easier, for example. This may aid both installation and removal of the gel sealant.

A further advantage of the pitted gel surface texture is that it may, at least in some circumstances, facilitate installation of the gel sealant at low temperatures (e.g. down to about -10°C).

The gel sealant object according to the invention preferably comprises a monolithic mass of gel sealant having a surface with the said pitted texture which is visible to the naked eye.

The pitted gel surface texture preferably comprises a substantially irregular arrangement of peaks and indentations. The maximum depth of the indentations of the pitted gel surface texture, as measured from the highest peak immediately adjacent to each indentation, is preferably no more than 5mm, more preferably no more than 4mm, even more preferably no more than 3mm, even more preferably no more than 2mm, especially no more than 1.5mm. Advantageously, at least 90% of the indentations of the pitted gel surface texture may have a depth of at least 0.5mm, preferably at least 0.75mm, as measured from the highest peak immediately adjacent to each indentation. It is generally preferred that the mean distance between the deepest point of adjacent indentations of the pitted gel surface texture is in the range 0.2mm to 3mm, more preferably in the range 0.5mm to 2mm.

In preferred embodiments of the invention, the ratio of the actual surface area of the pitted gel surface texture to the theoretical surface area of a substantially perfectly

smooth surface of the same (apparent) size is in the range 1.1:1 to 3:1, more preferably 1.2:1 to 2:1.

The pitted gel surface texture of objects according to the invention may advantageously be formed by moulding or casting the gel sealant on a surface which has a pitted, knobbly, undulating or rough surface texture.

Accordingly, a third aspect of the invention provides a method of making an object according to the second aspect of the invention, in which the pitted gel surface is formed by moulding or casting the gel sealant on a surface which has a pitted, knobbly, undulating or rough surface texture. The pitted, knobbly, undulating or rough surface on which the gel sealant has been moulded or cast may, for example, comprise an abrasive surface, preferably comprising abrasive particles adhered to a substrate. The abrasive surface preferably comprises the abrasive surface of glass-paper, sand-paper, emery-paper, emery-cloth, emery-board, or other abrader. The abrader is preferably of grade P20 or equivalent.

According to a fourth aspect, the invention provides a container for containing one or more electrical connections, electrical or electronic devices, optical fibres, or optical devices, the container including an object according to the second aspect of the invention, which object is adapted to seal at least part of the container and/or one or more connections, devices or fibres contained therein.

The gel material used as the sealant or article may, for example, comprise silicone gel, urea gel, urethane gel, thermoplastic gel, or any suitable gel or gelloid sealant material. Preferred gels comprise a liquid extended polymer composition.

Preferably the gel material has a hardness at room temperature as determined using a Stevens-Voland Texture Analyser of greater than 45g, particularly greater than 50g, especially greater than 55g, e.g. between 55g and 60g. It preferably has a stress-relaxation of less than 12%, particularly less than 10% and especially less than 8%. Ultimate elongation is preferably greater than 200%, more preferably greater than

500%, especially greater than 1000%, as determined according to ASTM D638. Tensile modulus at 100% strain is preferably at least 1.8 MPa more preferably at least 2.2 MPa. In general compression set is preferably less than 35%, more preferably less than 25%, especially less than 15%. Preferably, the gel material has a cone penetration as measured by ASTM D217 of at least 50 (10^{-1}mm), more preferably at least 100 (10^{-1}mm), even more preferably at least 200 (10^{-1}mm) and preferably no greater than 400 (10^{-1}mm), especially no greater than 350 (10^{-1}mm).

The extender liquids employed in preferred gel materials, for example those described in the aforementioned International Patent Applications, preferably comprise oils. The oils may be hydrocarbon oils, for example paraffinic or napthenic oils, synthetic oils for example polybutene or polypropene oils, or mixtures thereof. The preferred oils are mixtures of non-aromatic paraffins and naphthenic hydrocarbon oils. The gel may contain additives, e.g. such as moisture scavengers (e.g. Benzoyl chloride), antioxidants, pigments and/or fungicides.

Specific embodiments of the invention will now be described, by way of example, with reference to the accompanying drawings, in which:

Figure 1 is schematic illustration of a cross-section through an object according to the invention;

Figure 2 is a detail of the pitted gel surface texture of the object illustrated in Figure 1;

Figure 3 is a schematic illustration of a disc-shaped object according to the invention;

Figure 4 is an illustration of an example of a container according to the invention; and

Figure 5 is an illustration of a test sample of an object according to the invention.

Figure 1 shows, schematically, a cross-section of an object 1 comprising gel sealant, according to the invention. The object 1 has a gel surface 3 which has a pitted texture which is visible to the naked eye. The pitted gel surface texture 3 comprises a

substantially irregular arrangement of peaks 5 and indentations 7. The object 1 comprises a monolithic mass of gel sealant.

Figure 2 shows, also schematically, a detail of the pitted texture of the gel surface 3 of the object 1 shown in Figure 1. The figure shows how the depths, D1 and D2, of two indentations are defined. The depth of each indentation 7 is measured from the top T of the highest peak 5 immediately adjacent to the indentation, to the deepest point P of the indentation. The figure also shows how the distance L between adjacent indentations is defined. The distance L is defined as the distance between the deepest point P of adjacent indentations.

Figure 3 is a schematic illustration of a disc-shaped object 1 according to the invention. Other shapes are, of course possible. The object according to the invention may, for example, comprise a tape or a strip; an elongate object which may, for example, have a generally circular or other profile cross-section; a sheet; a block; or generally any shape suitable for sealing against an article or between articles.

Figure 4 shows an example of a container 9 according to the invention. The container 9 may, for example, contain an electrical connection, e.g. a telecommunications drop-wire splice. The container 9 has a circular cross-section cavity 11 which may be substantially filled, and thereby sealed, by the disc-shaped gel sealant object 1 illustrated in Figure 3. The object 1 may be inserted into the cavity 11 such that its pitted texture gel surface 3 contacts the electrical connector 13 in the cavity.

Figure 5 is an illustration of a test sample of an object 1 according to the invention. One half of the object, designated A, has a pitted gel surface texture 3 which is visible to the naked eye, according to the invention. The other half of the object, designated B, has (or at least had, before the test) a substantially smooth gel surface. A test block (not shown) having a regular pattern of square cross-section pegs was forced against both halves of the test sample for 12 hours at 60°C. Figure 5 shows the resulting effect on the gel surface. The half of the sample designated A (having the pitted surface texture of the invention) was substantially undamaged. The other half of

the test sample designated B contained deep cuts in the surface where the square pegs had dug into the gel sealant.

CLAIMS :

1. An article or object comprising a body of gel material having a deliberately roughened exterior surface.

2. An article according to claim 1, comprising melt-extruded thermoplastic gel having a tack-reducing degree of surface melt fracture.

3. An article according to claim 2, wherein the degree of surface melt fracture is at least sufficient to provide the article with a visually-observable matt surface finish.

4. An article according to claim 3, having a sharkskin melt-fractured surface finish.

5. A method of making a melt-extruded thermoplastic gel article, wherein, during the melt extrusion process, surface melt fracture of the gel is induced to a degree which reduces the tack of the finished article.

6. A method according to claim 5, wherein the surface melt fracture is induced to a degree at least sufficient to provide the finished article with a visually-observable matt surface finish.

7. A method according to claim 6, wherein the surface melt fracture is induced to a degree which provides the finished article with a sharkskin melt-fractured surface finish.

8. An object according to claim 1, comprising gel sealant, a gel surface of which has a pitted texture which is visible to the naked eye.

9. An object according to Claim 8, which comprises a monolithic mass of gel sealant.

10. An object according to Claim 8 or Claim 9, in which the pitted gel surface texture comprises a substantially irregular arrangement of peaks and indentations.

11. An object according to Claim 10, in which the maximum depth of the indentations of the pitted gel surface texture, as measured from the highest peak immediately adjacent to each indentation, is no more than 5mm, preferably no more than 4mm, more preferably no more than 3mm, even more preferably no more than 2mm, especially no more than 1.5mm.

12. An object according to Claim 10 or Claim 11, in which at least 90% of the indentations of the pitted gel surface texture have a depth of at least 0.5mm, preferably at least 0.75mm, as measured from the highest peak immediately adjacent to each indentation.

13. An object according to any one of claims 10 to 12, in which the mean distance between the deepest point of adjacent indentations of the pitted gel surface texture is in the range 0.2mm to 3mm, preferably in the range 0.5mm to 2mm.

14. An object according to any of claims 8 to 13, in which the ratio of the actual surface area of the pitted gel surface texture to the theoretical surface area of a substantially perfectly smooth surface of the same apparent size is in the range 1.1:1 to 3:1, preferably 1.2:1 to 2:1.

15. An object according to any of claims 8 to 14, in which the pitted gel surface texture has been formed by moulding or casting the gel sealant on a surface which has a pitted, knobbly, undulating or rough surface texture.

16. A method of making an object according to any of claims 8 to 15, in which the pitted gel surface texture is formed by moulding or casting the gel sealant on a surface which has a pitted, knobbly, undulating or rough surface texture.

17. An object according to Claim 16, or a method according to Claim 9, in which the pitted, knobbly, undulating or rough surface on which the gel sealant has been moulded or cast comprises an abrasive surface, preferably comprising abrasive particles adhered to a substrate.

18. An object or method according to Claim 17, in which the abrasive surface comprises the abrasive surface of glass-paper, sand-paper, emery-paper, emery-cloth, emery-board, or other abrader.

19. An object or method according to Claim 118, in which the abrader is of grade P20 or equivalent.

20. A container for containing one or more electrical connections, electrical or electronic devices, optical fibres, or optical devices, the container including an object according to any one of claims 8 to 19, which object is adapted to seal at least part of the container and/or one or more connections, devices or fibres contained therein.

21. An article or method according to any preceding claim, wherein the melt-fractured gel comprises a [hard block-elastomeric block-hard block] triblock copolymer whose elastomeric mid-block is extended with at least 200 parts by weight of extender fluid per 100 parts by weight of the triblock copolymer.

22. An article or method according to claim 21, wherein the triblock copolymer is a [styrene-hydrogenated alkylene-styrene] triblock copolymer, preferably having a hydrogenated alkylene mid-block comprising ethylene/propylene and/or ethylene/butylene units.

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Fig.1.

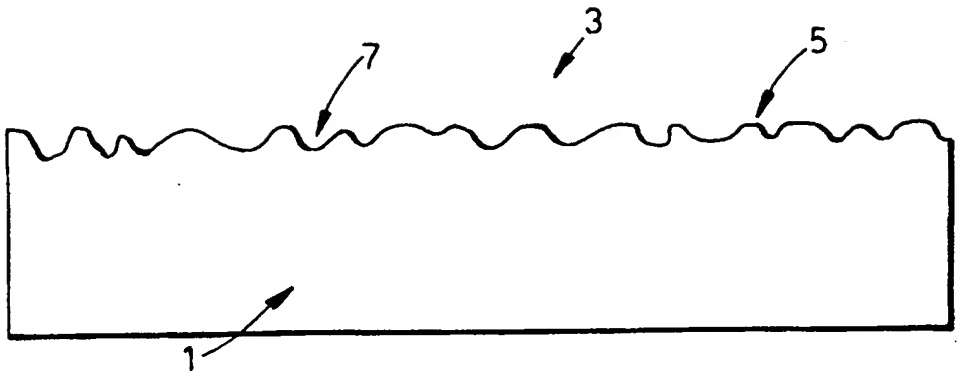


Fig.2.

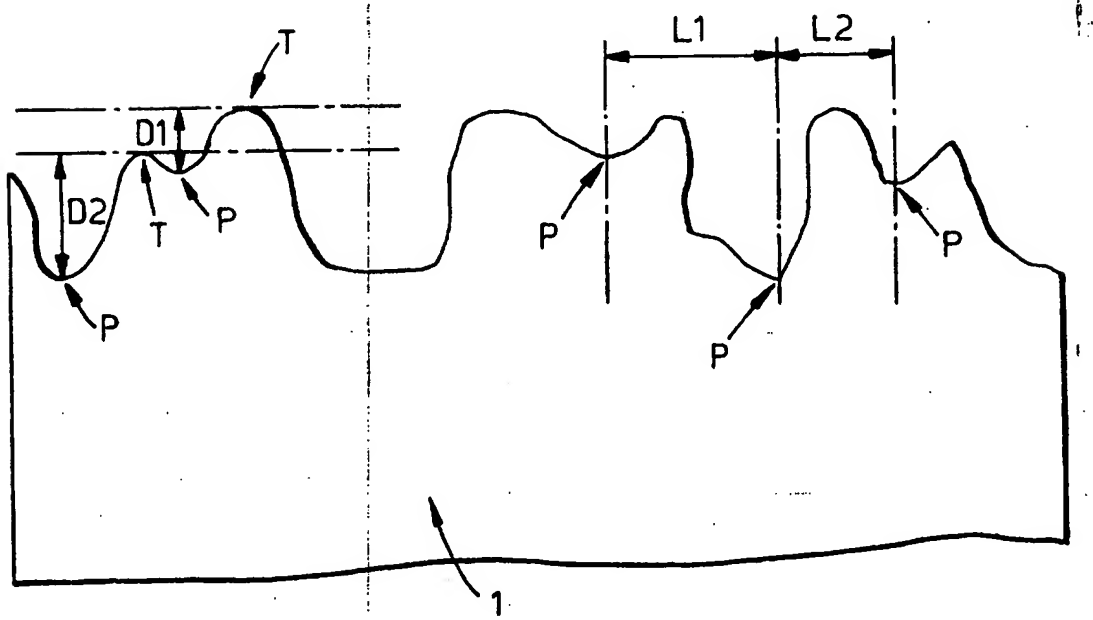


Fig.3.

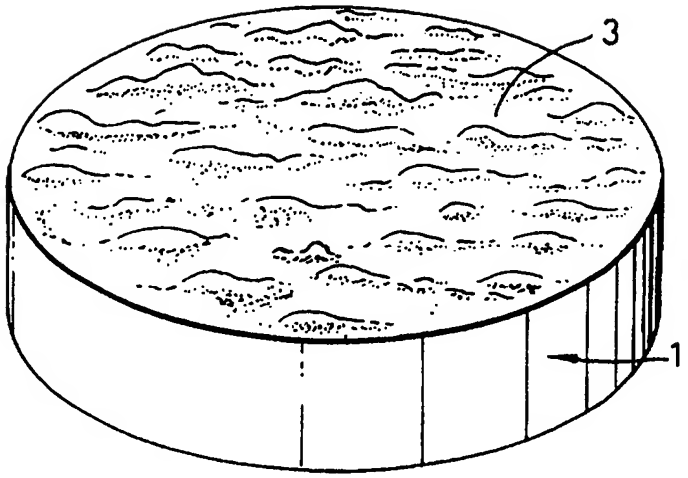


Fig.4.

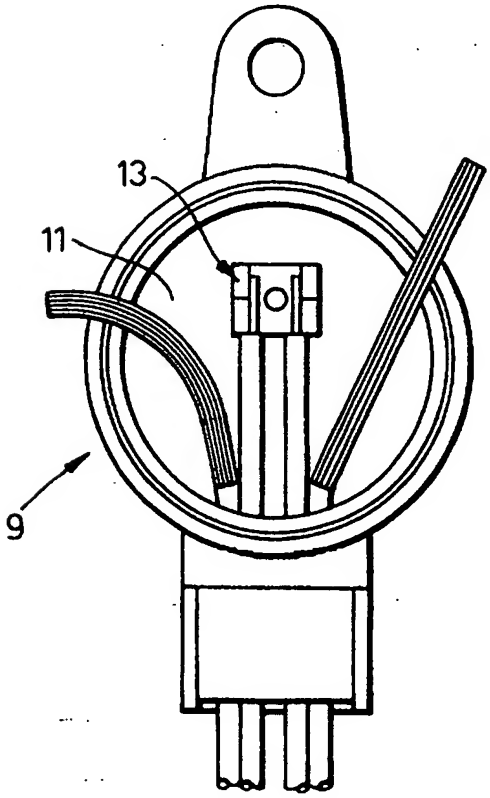
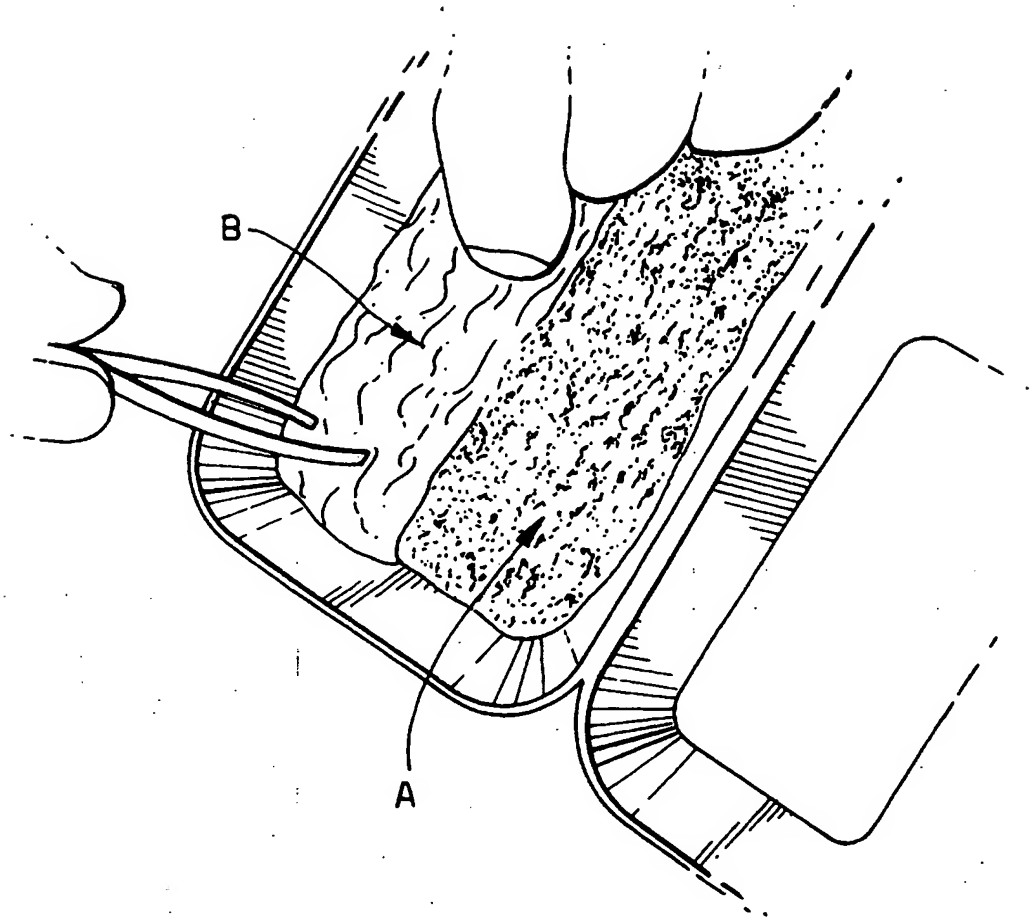


Fig.5.



Internat l Application No
PCT/GB 96/02871

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C09K3/10 B29C59/02 C08J3/075 H01B7/28 H02G15/08

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C09K B29C C08J H01B H02G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	see page 7, line 24 - line 27 see page 13, line 52 - line 56 see page 14, line 19 - line 23 see figures; examples	3,4
A	WO 93 23472 A (RAYCHEM LTD ;HAMMOND PHILIP JAMES (GB); HUDSON JOHN MICHAEL (GB);) 25 November 1993 cited in the application see the whole document	1,8,9, 21,22
A	WO 94 18273 A (RAYCHEM LTD ;HAMMOND PHILIP JAMES (GB); HUDSON JOHN MICHAEL (GB);) 18 August 1994 cited in the application see the whole document	1,8,9, 21,22

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
12 March 1997	21.03.97

Name and mailing address of the ISA	Authorized officer
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Form PCT/ISA/210 (second sheet) (July 1992)

Internat l Application No
PCT/GB 96/02871

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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